

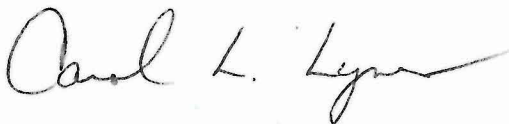
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE: **January 11, 2008**

JAN 13 PM 12:08

SUBJECT: Tissue Chemical Data Assessment - Sampling and Testing of Material Proposed for Dredging from the San Juan Harbor, Puerto Rico.

FROM: Carol L. Lynes, Environmental Scientist
Monitoring and Assessment Branch



TO: Mark Reiss, Environmental Scientist
Dredging Sediments and Ocean Team

As per your request, I have completed my assessment of the tissue chemical data summaries produced for the San Juan Harbor Dredging Project. Specific review criteria and comments are included in the attached chemical data assessment report.

The assessment of the chemical data associated with this project concludes that the data are "acceptable" and "acceptable with condition". Details of the data acceptability determinations are provided in the attached report narrative and tables.

If you have any questions, please feel free to contact me at 732-321-6760 or lynes.carol@epa.gov.

Attachment

Cc. J. Ferretti, DESA-LAB

2000 JAN 15 PM 1:07
U.S. EPA-REGION 2
CLEANWATER REGULATORY BR.

SUMMARY OF FINDINGS AND TECHNICAL RECOMMENDATIONS - CHEMICAL TESTING

Project(s): Dredged Material Testing for Proposed Ocean Disposal – San Juan Harbor, Puerto Rico
Chemical Testing Laboratories: Battelle Laboratories

Chemical testing was conducted on tissue matrices for PAH Compounds, Pesticides, PCB Congeners, and Metals. Tissue summary data, including quality control results, were reviewed for compliance with the established testing requirements as specified below.

Tissue samples

Procedural/Method Blank - One per batch of 1-20 samples

Matrix spike (in Triplicate) - Fortified 3 to 5x the RL identified in the manual; one set per batch of 1-20 samples

LCS/LFB - Fortified to 3 to 5x the RL identified in the manual; one per batch of 1-20 samples

SRM - Not required

Surrogate Spike - per sample (Organics Only)

Initial Calibration Check Samples with calculated results of the recoveries.

Continuing Calibration Check Samples with calculated results of the recoveries.

The assessment and conclusions on data acceptability are outlined in the following tables. The acceptability of the data for each chemical group and matrix is categorized as "acceptable", "acceptable with condition", "unacceptable", and/or "not determined". It is our opinion that the data with an "acceptable" or "acceptable with condition" may be used; however, if indicated, please note the particular condition. The category "not determined" is used if insufficient data were provided.

Reporting Comments:

For future submittals, Anamar must include copies of the analytical results data sheets for all samples, including the quality control samples.

Laboratory Analysis Comments:

Pesticides/PCBs – The initial calibration check samples for all analytical batches did not meet the established acceptance criterion.

07-0043 Congener 206 exceeded the criterion.

07-0048 Congeners 206 and 209 exceeded the criterion.

07-0049 Congeners 206 and 209 exceeded the criterion.

PAHs – The matrix spike, matrix spike duplicate, and matrix spike triplicate samples were over-fortified in all analytical batches. Over-fortified spike samples will not be accepted.

Metals (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)

<u>QC Measurement</u>	<u>Frequency</u>	<u>Calculation</u>	<u>QC Control Acceptance Criteria</u>	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery And %RSD	70 -130% (Recovery) 30% (RSD)	<i>M. nasuta</i> : All results are within the acceptance criteria. <i>N. virens</i> : Zn was recovered below 70% in the MS and the MST. The low recovery resulted in an RSD exceeding 30%. All remaining results are within the acceptance criteria.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	% Recovery *Evaluated for analytes > 3x RL	70 - 130%	<i>M. nasuta</i> : Not required for tissue analysis. <i>N. virens</i> : Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery	70 -130%	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	90 - 110%	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Continuing Calibration Checks using Calibration Standards	Minimum – check calibration at middle and end of each batch or 1 per 10 analyses, which ever is greater	% Recovery	90 - 110% from initial calibration for each analyte	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Data Acceptability	<i>M. nasuta</i> : Acceptable. <i>N. virens</i> : Acceptable with condition that the results for zinc are biased low.			

Organics (Pesticides, PCBs) Analytical Batch: 07-0043

<u>QC Measurement</u>	<u>Frequency</u>	<u>Calculation</u>	<u>QC Control Acceptance Criteria</u>	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	<i>N. virens</i> : All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	<i>N. virens</i> : All results are within the acceptance criteria.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	<i>N. virens</i> : Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	<i>N. virens</i> : All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	<i>N. virens</i> : All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	<i>N. virens</i> : Congener 206 exceeds the criteria. The bias for this congener is unknown.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	<i>N. virens</i> : All results are within the acceptance criterion.
Data Acceptability	<i>N. virens</i> : Acceptable with the condition that the result for congener 206 is to be considered estimated. All remaining congeners are acceptable.			

NOTE: The criteria are for each analyte

Organics (PAHs)

Analytical Batch: 07-0043

<u>QC Measurement</u>	<u>Frequency</u>	<u>Calculation</u>	<u>QC Control Acceptance Criteria</u>	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	<i>N. virens</i> : All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	<i>N. virens</i> : The spiked samples were over-fortified. The MS, MSD, MST samples are not acceptable, even though all of the results are within the acceptance criterion. Over-fortified spike samples will not be accepted.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	<i>N. virens</i> : Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	<i>N. virens</i> : All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	<i>N. virens</i> : All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	<i>N. virens</i> : All results are within the acceptance criterion.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	<i>N. virens</i> : All results are within the acceptance criterion.
Data Acceptability	<i>N. virens</i> : Acceptable.			

NOTE: The criteria are for each analyte

Organics (Pesticides, PCBs) Analytical Batch: 07-0048

<u>QC Measurement</u>	<u>Frequency</u>	<u>Calculation</u>	<u>QC Control Acceptance Criteria</u>	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	<i>M. nasuta</i> : All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	<i>M. nasuta</i> : All results are within the acceptance criteria.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	<i>M. nasuta</i> : Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	<i>M. nasuta</i> : All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	<i>M. nasuta</i> : All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	<i>M. nasuta</i> : Congeners 206 and 209 exceed the criteria. The bias for these congeners is unknown.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	<i>M. nasuta</i> : All results are within the acceptance criterion.
Data Acceptability	<i>M. nasuta</i> : Acceptable with the condition that the results for congeners 206 and 209 are to be considered estimates. All remaining congeners are acceptable.			

NOTE: The criteria are for each analyte

Organics (PAHs)

Analytical Batch: 07-0048

<u>QC Measurement</u>	<u>Frequency</u>	<u>Calculation</u>	<u>QC Control Acceptance Criteria</u>	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	<i>M. nasuta</i> : All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	<i>M. nasuta</i> : The spiked samples were over-fortified. The MS, MSD, MST samples are not acceptable, even though all of the results are within the acceptance criterion. Over-fortified spike samples will not be accepted.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	<i>M. nasuta</i> : Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	<i>M. nasuta</i> : All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	<i>M. nasuta</i> : All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	<i>M. nasuta</i> : All results are within the acceptance criterion.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	<i>M. nasuta</i> : All results are within the acceptance criterion.
Data Acceptability	<i>M. nasuta</i> : Acceptable.			

NOTE: The criteria are for each analyte

Organics (Pesticides, PCBs) Analytical Batch: 07-0049

<u>QC Measurement</u>	<u>Frequency</u>	<u>Calculation</u>	<u>QC Control Acceptance Criteria</u>	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	<i>M. nasuta</i> : All results are within the acceptance criteria. <i>N. virens</i> : All results are within the acceptance criteria.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	<i>M. nasuta</i> : Not required for tissue analysis. <i>N. virens</i> : Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	<i>M. nasuta</i> / <i>N. virens</i> : Congeners 206 and 209 exceed the criteria. The bias for these congeners is unknown.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	<i>M. nasuta</i> / <i>N. virens</i> : All results are within the acceptance criterion.
Data Acceptability	<i>M. nasuta</i> : Acceptable with the condition that the results for congeners 206 and 209 are to be considered estimates. All remaining congeners are acceptable. <i>N. virens</i> : Acceptable with the condition that the results for congener 206 and 209 are to be considered estimates. All remaining congeners are acceptable.			

NOTE: The criteria are for each analyte

Organics (PAHs) Analytical Batch: 07-0049

<u>QC Measurement</u>	<u>Frequency</u>	<u>Calculation</u>	<u>QC Control Acceptance Criteria</u>	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	<i>M. nasuta</i> / <i>N. virens</i> : The spiked samples were over-fortified. The MS, MSD, MST samples are not acceptable, even though all of the results are within the acceptance criterion. Over-fortified spike samples will not be accepted.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	<i>M. nasuta</i> : Not required for tissue analysis. <i>N. virens</i> : Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	<i>M. nasuta</i> : All results are within the acceptance criterion. <i>N. virens</i> : All results are within the acceptance criterion.
Data Acceptability	<i>M. nasuta</i> : Acceptable. <i>N. virens</i> : Acceptable.			

NOTE: The criteria are for each analyte